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THE OFFICE OF RESEARCH



OFFICE OF THE DIRECTOR

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TO:

Office of Grants and Contracts

Attention: Code SC

National Aeronautics and Space Administration

Washington, D. C. 20546

SUBJECT:

Progress Report

on

"INVESTIGATION OF THE SOLIDIFICATION, STRUCTURE AND PROPERTIES OF EUTECTIC ALLOYS INCLUDING CONSIDERATION OF PROPERTIES CONTROL"

NASA Research Grant NGR-39-007-007 Lehigh University Acct. No. 1056-65

Submitted by:

DATE:

June 1969

R. W. Kraft, Professor

R. W. Hertzberg, Associate Professor

Co-investigator

INVESTIGATION OF THE SOLIDIFICATION, STRUCTURE AND PROPERTIES OF EUTECTIC ALLOYS

This report supplements the information contained in various technical papers written under auspices of this grant and forwarded to NASA headquarters as they became available. It consists of the following sections:

- A. Work completed.
- B. Research in progress.
- C. Administrative matters.

A. WORK COMPLETED

Thesis research wholly included under this grant and completed some time ago but only recently made available as reprints of technical papers include the following:

- W. R. Hoover and R. W. Hertzberg, "The Fatigue Characteristics of Unidirectionally Solidified Al-Al₃Ni Eutectic Alloy," <u>Trans. ASM</u>, Vol. 61, No. 4, Dec. 1968, pp. 769-776.
- R. T. Quinn, R. W. Kraft and R. W. Hertzberg, "Structure and Elevated-Temperature Mechanical Behavior of Unidirectionally Solidified Ni-Ni3Nb Eutectic Alloy," <u>Trans. ASM</u>, Vol. 62, No. 1, March 1969, pp. 38-44.

In addition other research for which NASA assisted via faculty participation and by providing necessary supplies is as follows:

- K. D. Sheffler, R. W. Kraft and R. W. Hertzberg, "Microstructure and Crystallography of the Ni-Ni₃Ti Eutectic Alloy," <u>Trans. Met. Soc. AIME</u>, Vol. 245, Feb. 1969, pp. 227-231.
- K. D. Sheffler, R. W. Hertzberg and R. W. Kraft, "Elevated Temperature Mechanical Properties and Fracture Behavior of a Ni-Ni₃Ti Eutectic Alloy," Trans. ASM, Vol. 62, No. 1, March 1969, pp. 105-116.

As is evident from the titles of these papers, a major portion of the NASA sponsored research on eutectic alloys at Lehigh has been concerned with exploring the potential of eutectic composites as structural materials at elevated temperatures or under cyclic loading conditions. Investigation of mechanical properties continues to be a principal thrust of our effort as described in the next section. Progress on the other phase of our work on physical properties is discussed in the last section on administrative matters.

B. RESEARCH IN PROGRESS

I. Fatigue in Ni-Ni₃Nb

W. R. Hoover, a graduate assistant, is continuing his research on the fatigue behavior of composite eutectics. Attention is being focused on the Ni-Ni₃Nb system since Quinn's work (see above) has shown that the material, or a modification thereof--Ni₃Al-Ni₃Nb as shown by Thompson and Lemkey also in the March 1969 issue of Trans. ASM--has great interest as a structural material. Work currently under way is discussed under the headings solidification studies, mechanical behavior of Ni₃Nb and plans for the future.

Solidification Studies

Modifications of the vertical melting apparatus located in the Materials Research Center have been completed, thereby, allowing us to unidirectionally solidify in a vertical direction ingots of the Ni-Ni₃Nb eutectic alloy. The first solidification experiments have met with considerable success. The microstructural alignment in these ingots is vastly superior to that previously obtained in horizontally grown ingots where the platelets were up to twenty (20) degrees off the ingot growth axis (Figure 1). Theoretical predictions and experimental findings in other composite systems indicate that improvement in mechanical properties should be observed in the vertically grown ingots as a result of the improved microstructural alignment.

Master heats of entectic composition have been prepared. Initial experiments will focus on optimization of the solidification parameters leading to the attainment of the highest quality microstructures. When this exploratory phase has been completed, ingots will be routinely produced for subsequent mechanical property evaluation.

Mechanical Behavior of NiaNb

In order to more completely understand the mechanical response of the Ni-Ni₃Nb eutectic, knowledge of the deformation and fracture behavior of both phases is required. To this end, considerable effort has been devoted to an analysis of the mechanical response of the reinforcing, Ni₃Nb phase.

Large grained ingots of the Ni₃Nb phase were prepared and cut into small bend specimens. Three surfaces of each bend specimen were metallographically polished and etched with a 35% HNO₂, 2%HF and 68%H₂O solution. The crystallographic orientation of several large grains in the bend specimens were determined by Laue X-ray diffraction techniques. After bending, two surface and one surface trace analyses were performed with the observed slip and twinning markings. To more clearly reveal details of the Ni₃Nb grain boundaries, twin boundaries and slip offset markings, optical examination was done under polarized light.

Twinning: After bending, the specimens were repolished to eliminate slip traces. Several sets of parallel markings were retained after repolishing (Figure 2) and analyses revealed two operative twin modes: {011} and two varients of {112} type twins. The {011} twins were observed to be rather wide implying a relatively small twinning shear. A second varient of the {011} twins was observed only on rare occasions. The {112} twins with the regularly observed second varient were always narrow suggesting a large twin shear. The Ni₃Nb alloy was found to crack preferentially along the matrix-twin interface of the {112} twins which is in accord with the belief that the narrow {112} twins have a rather large twinning shear.

Slip: Examination of the prepolished bend specimens revealed the presence of two slip systems. The presence of slip markings was observed relatively rarely.

Efforts to obtain two surface slip data have, thus far, been unsuccessful but one surface trace analysis has been conducted. These data are insufficient for slip plane identification at the present time. Present plans call for the production of more Ni₃Nb ingots in this phase of the investigation. Trace analyses will be conducted on slip markings from compression specimens. Previous work conducted under this contract (R. T. Quinn) indicates that compression testing may produce more slip and less twinning in the Ni₃Nb phase for certain loading directions.

Fracture of Ni₃Nb: An initial attempt to employ the new acquired ARL Electron Microprobe with a scanning attachment in fractographic analysis has been successful. Fracture Ni₃Nb bend specimens have been examined and the usefulness of the scanning electron microscope as a fractographic tool has been established, particularly at intermediate magnifications where fracture surface topology is of interest. For example, Figure 3a shows the entire fracture surface of a Ni₃Nb bend specimen at a low magnification; failure was believed to have initiated at a (112) twin-matrix interface characterized by the flat, featureless area shown in the lower right corner of the photograph. After encountering either another twin or a grain boundary, the crack proceeded through the remainder of the specimen. The topology developed on the fracture surface when the crack intersected other twins is shown in Figure 3b.

As can be seen from the above discussion and included photographs, the scanning electron microscope capability of the electron microprobe can provide valuable topographic information within limits of the instrument resolution capacity, approximately 2000Å.

Plans for the Future

Experimental studies with the Ni-Ni₃Nb eutectic alloy will proceed in several directions now that much of the necessary background work has been completed. Further studies of the slip systems in Ni₃Nb will be conducted along with analysis

of the multiple twinning systems. Specimens of the Ni-Ni₃Nb eutectic alloy will be unidirectionally solidified in the vertical furnace. Initial mechanical testing will be conducted to evaluate tensile properties of the alloy with the superior microstructural alignment. In addition, determination of elastic modulus of the two phase structure will be attempted. If time permits in the next report period, initial fatigue tests will be performed. The determination of fracture mechanisms of this eutectic alloy will continue with the aid of the scanning electron microscope and electron fractographic techniques.

II. Mechanical Behavior of Mg-Mg2Ni

As described in the Proposal for Continuation of Grant NGR-39-007-007, several new experimental programs were suggested. Mr. Kenneth Eckelmeyer, recipient of an industrial fellowship, is now addressing himself to a study of the Mg-Mg2Ni eutectic alloy. Since his fellowship yields extremely limited funds for research expenses (supplies, service charges, etc.) some funds from the present grant are being used to assist him.

This research program focuses on the mechanical behavior of controlled eutectic alloys based on light weight hexagonal close packed metals. The outstanding potential of such materials has recently been demonstrated in a survey of several magnesium based controlled eutectics. The particularly promising Mg-Mg₂Ni composite exhibited excellent tensile and compressive strengths (54,800 psi and 77,500 psi respectively) even though a 100% aligned structure was not obtained.

The present program will be a detailed investigation of the mechanical behavior of the unidirectionally solidified Mg-Mg₂Ni eutectic. It is anticipated that modifications in the solidification procedure will result in a more perfectly aligned structure having still better properties. After the best possible structure has been obtained an extensive testing program will be carried out. This will include tensile, compresive, and fatigue tests at room temperature as well as some elevated temperature property measurements. In addition to the testing program, a fundamental

study of the nature of deformation and fracture will be undertaken. X-ray diffraction and metallographic techniques will be applied to determine how the crystallographic and morphological relationships between the two phases effect the mechanisms of elastic and plastic deformation. Fracture processes will be studied by optical metallography and electron fractography. It is expected that this investigation will provide mechanical property data on a promising light weight composite as well as an insight into the fundamental behavior and potential of aligned eutectics with HCP matrices.

Progress so far has consisted primarily of extensive modification of the unidirectional solidification apparatus. While this work has been time consuming, it should pay dividends in the form of a better aligned structure than could be obtained on the old equipment. Master heats of the Mg-Mg₂Ni eutectic alloy are presently being prepared. Unidirectional solidification work will begin in the very near future.

C. ADMINISTRATIVE MATTERS

Professor R. Wayne Kraft continues as principal investigator in the field of solidification and eutectic structures and as chief administrative officer on this grant. Associate Professor R. W. Hertzberg continues as co-investigator specializing in the area of mechanical behavior and deformation. Until 31 January 1969, Assistant Professor Richard N. Tauber served as co-investigator with primary responsibility in the area of physical properties. At that time, he resigned from Lehigh to accept a position in industry. Beginning 1 June 1969, Associate Professor Walter C. Hahn took up these duties and is presently planning work in this area, which, quite frankly has not progressed as much as we would have liked in the past year.

Mr. William R. Hoover continues to serve as a graduate assistant on this grant and is working with Professors Hertzberg and Kraft on the program concerned with fatigue in the Ni-Ni₃Nb system. He has completed all requirements for the Ph.D. degree save his thesis research; it is anticipated that this will be completed by June 1970. Mr.

Kenneth Eckelmeyer is not drawing a stipend from the grant but is drawing upon it for supplies and operating expenses. His Ph.D. program is not as far advanced as that of Mr. Hoover although he has completed his master's program.

For over a year the principal investigator has been trying to obtain a research assistant to work in the area of physical properties. The draft situation has been directly responsible for the fact that the vacancy is not yet filled. Files at Lehigh can attest that three capable candidates turned down our offers to work in this area for that reason. Nevertheless the problem appears to have been solved. A candidate not subject to the draft has been accepted for admission to the graduate school and will commence working in this area with Professors Hahn and Kraft on or about 1 September 1969.

The three faculty investigators participating in this research hope to continue working in the area. They have several ideas for worthwhile research which builds on that already completed on this and other grants and on that reported in the literature. A renewal proposal at approximately the same rate of expenditure (approximately \$39,000/yr.) is now in the final stages of negotiation.



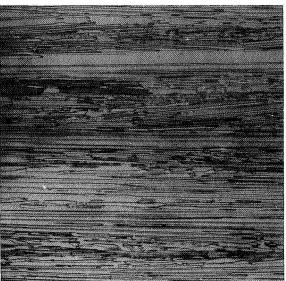


Figure 1: Photomicrographs Showing Improved Alignment in Ni-Ni Nb Microstructure. Vertical Direction is Growth Axis. a) Grown Horizontally; b) Crown Vertically. Magnification: 125X

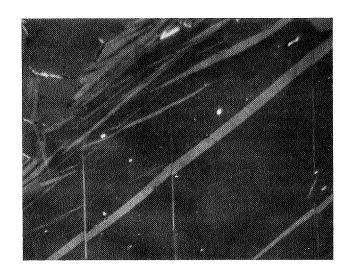
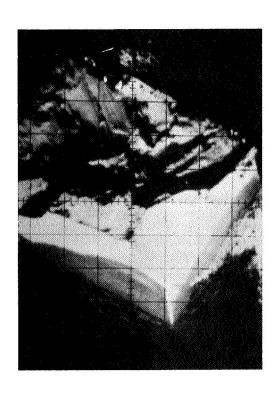


Figure 2: Photomicrograph of Ni₃Nb Phase Revealing Two Types of Twins. Polarized Light. Magnification: 200X.



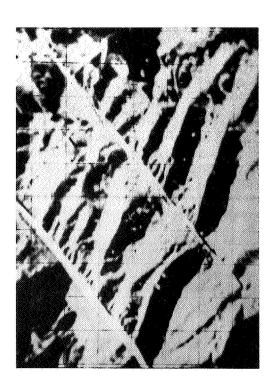


Figure 3: Scanning Electron Fractographs of Fractured Ni₃Nb Bend Specimen. a) Magnification: 20X; b) Magnification: 350X.